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Prepared for
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U.S. DEPARTMENT OF COMMERCE
Robert Mosbacher, Secretary
NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY
Raymond G. Kammer, Acting Director

INTRODUCTION:

The Naval Ordnance Laboratory at Indian Head, Maryland identified certain head caps used on missile weapons systems as potentially susceptible to failure due to suspected improper heat treatment. The suspect head caps were reported to have been fabricated from a cast 17-4 PH stainless steel alloy. The design specification for the head caps required that they be heat treated according to the Society for Automotive Engineers (SAE) Aerospace Material Specification AMS-5355D, condition H1000 (1).

Production records and test results were not complete and/or showed that the head caps did not meet the heat treatment specification. The Metallurgy Division at the NIST was asked to evaluate selected head caps to determine if they had been heat treated in compliance with AMS-5355D, condition H1000.

MATERIALS:

Four head caps selected by personnel at the Naval Ordnance Laboratory were obtained by the NIST. All four head caps were similar in size and shape to that shown in Figure 1. The four head caps were reported to be from different, unidentified sources or heat treatment lots. The head caps were identified by code number as 5066, 5441, 20, and NIST 1.

The chemical composition and hardness required by specification AMS-5355D are shown in Table 1 and the heat treatments required by this specification are shown in Table 2.

EXPERIMENTAL PROCEDURE:

For each of the four as-received head caps, chemical composition was determined, hardness measurements were made on full sections (shown in Figure 1), and the microstructure was determined by optical metallographic procedures. Sections, each approximately one half inch in thickness by two inches in length, were cut from head caps numbered 5066 and 5441. These sections were heat treated by homogenization, solution treatment, and varying ageing treatments as specified in the literature (2) and in AMS specification 5355D. Microstructural evaluations and hardness measurements were made on these sections after each heat treatment.

Homogenization of the 17-4 PH sections consisted of heating the specimens to 2150°F ± 25°F for two hours and one-quarter hours followed by air cooling to below 70°F. The homogenizing heat treatment was carried out in a Lindberg/HeviDuty tube furnace. No protective atmosphere was used. Prior to placing the specimens into the furnace, the furnace was set at the homogenizing treatment temperature of 2150°F, and the temperature checked with a chromel/alumel thermocouple. The thermocouple was inserted to a depth of about one inch into the specimen. The temperature was monitored with a Doric Trendicator 400A Type K/°F unit. After cooling to room temperature, the specimens were immersed into room temperature tap water (less than 80°F) for at least than two hours in order to complete transformation.

The specimens were then solution annealed at 1900°F ± 25°F for one hour and air cooled to below 70°F. The solution heat treatment was carried out in the same furnace under the same controls and conditions as used in the homogenizing treatment.

Following the solution annealing, eight specimens,(two at each temperature), were aged at 925°F, 1025°F, 1075°F, and 1150°F for four hours and then air cooled. The ageing treatments were performed in a Lindberg air tempering furnace, the temperature of which was monitored with a thermocouple and a Doric Trendicator 400A Type K/°F unit.

Even though the ageing time was greater than that recommended in the specification, the literature (3) reports that ageing for this extra time has little effect on properties. Following each heat treatment, hardness measurements and photomicrographs of the resulting microstructures were taken. The homogenization, the solution, and the ageing treatments are shown in Table 3.

In addition to the heat treatments outlined above, heat treatments were also conducted on samples removed from as-received head caps 5066 and 5441. In these experiments, the samples were not given a homogenizing treatment, but were solution annealed and aged at 925°F and 1025°F.

EXPERIMENTAL RESULTS:

As-received Head Caps Samples:

The chemical compositions (duplicate determinations) for the as-received head caps 5066, 5441, NIST 1, and 20 are shown in Table 4 along with the composition given in specification AMS 5355D. The method used was multi-element atomic emission spectrometry, and is covered by ASTM Test Method E2 SM 9-46, Optical Emission Vacuum Spectrometric Analysis of Chromium-Bearing Ferrous Alloys.

The chemical analyses for all four head caps are in agreement with the specification with the exception of the silicon in head cap NIST 1, which was below the specified range.

Microstructural examinations were conducted on the as-received head caps. Figures 2 ,3 and 4 show photomicrographs of the as-received microstructures for head caps 5066, 5441, and 20 respectively. The microstructures consisted of elongated pools of ferrite (light grey constituent) dispersed in a matrix of martensite (dark constituent). The coarse pattern appears to be representative of an as-cast microstructure.

Figure 5 shows the microstructure for as-received head cap NIST 1. The microstructure was totally different from that for the as-received head caps 5066, 5441, or 20. The elongated pools of ferrite are not obvious and the martensite has taken on a different morphology. The fine microstructure observed in as-received head cap NIST 1 is not representative of an as-cast microstructure.

A hardness survey was taken over the entire cross-section of the as-received head caps. The results are shown in Figures 6, 7, 8, and 9. The hardness results indicated that head caps 5066, 5441, and NIST 1 had essentially similar average hardness values, HRC 29.6 to 31.5. By comparison, the average hardness for head cap 20 was significantly lower, HRB 98.8, which corresponds to an HRC of about 20.

For the as-received head caps, although 5066, 5441, and 20 appeared to have the same microstructure, the hardness of head cap 20 was substantially less than that of head cap 5066 or 5441. Although the microstructure of head cap NIST 1 appeared to be different than that of head caps 5066 and 5441, the average hardness for head cap NIST 1 (HRC 29.6) was essentially the same as that for head caps 5066 and 5441. The average hardness for head caps 5066 and 5441 was HRC 31.5 and HRC 27.8 respectively.

Homogenized, Solution Annealed, and Aged Samples:

Figures 10a and 10b show the microstructures obtained for samples sectioned from head caps 5066 and 5441 after homogenizing at 2150°F for two and one-quarter hours and air cooled. The microstructure consisted of regions of untempered martensite with plates of ferrite.

Figures 11a and 11b show the microstructures for samples taken from head caps 5066 and 5441 that were homogenized and then solution annealed at 1900°F for one hour and air cooled. After solution annealing, the microstructure was similar to the as-homogenized microstructure, e.g. untempered martensite and plates of ferrite. However, the microstructure after solution annealing was finer than that following homogenizing alone.

The hardness for the homogenized and solution annealed sample from head cap 5066 was HRC 36.8 compared with a hardness of HRC 35.8 after homogenizing, but before solution annealing. The hardness for the homogenized and solution annealed sample from head cap 5441 was HRC 31.8 compared with a hardness of HRC 37.7 after homogenizing, but before solution annealing. The hardness values are representative of 17-4 PH steel in the solution annealed condition (4).

Figures 12, 13, and 14 show the microstructures for samples taken from head caps 5066 and 5441 after they were homogenized, solution annealed, and then aged at 925°F, 1025°F, and 1075°F respectively. During the ageing treatment, copper-rich precipitates form (4). However in the ageing treatment done here, the precipitates were not large enough to be visible even after ageing at 1150°F.

Figures 15a and 15b show the microstructures observed after additional samples, also taken from head caps 5066 and 5441, were homogenized, solution annealed, but aged at 1150°F. The photomicrographs reveal that the microstructures were entirely different than those observed after the samples were similarly homogenized, solution annealed, and aged at 925°F, 1025°F, or 1075°F. Ageing at 1150°F caused the solution annealed microstructure to transform to a microstructure similar to, but not the same as the microstructure observed in the as-received head caps. The

microstructure consisted of elongated pools of ferrite and tempered martensite in contrast to the matrix of martensite and ferrite platelets observed at the lower ageing temperatures.

A hardness survey was conducted on samples taken from head caps 5066 and 5441 that were homogenized, solution annealed, and then aged at 925°F, 1025°F, 1075°F, and 1150°F. The results are shown in Table 5 and plotted in Figures 16 and 17.

A comparison of Figures 16 and 17 shows that the as-received hardness for head caps 5066 and 5441 were essentially the same, HRC 29.4 to 28.0 respectively. Homogenizing increased the hardness to HRC 35.8 and 37.7 respectively. The response to ageing was also essentially the same for both head caps in that the curves over the ageing temperature range of 925°F to 1150°F were almost identical, Figures 16 and 17. Only for the solution annealed samples was there any significant difference in the hardness values.

Solution Annealed and Aged Samples:

These samples were not given a homogenizing treatment prior to the solution anneal or ageing treatments. Figures 18a and 18b show the microstructures observed for samples taken from as-received head caps 5066 and 5441 after they were solution treated at 1900°F for one hour and then air cooled. The microstructures consist of areas of untempered martensite (dark) and long islands of ferrite. Figures 19a and 19b show the microstructures for these same samples after they were aged at 925°F. The microstructures were not similar to specimens taken from heat caps 5066 and 5441 that were homogenized, solution annealed, and aged at 925°F for four hours.

The solution treated and the aged hardness values are shown in Table 5. The solution annealed hardness values for head caps 5066 and 5441 were HRC 30.8 and HRC 36.4, respectively. After ageing at 925°F, the hardness increased to HRC 38.0 for head cap 5066, and to HRC 39.3 for head cap 5441.

DISCUSSION:

A metallurgical investigation was conducted on four 17-4 PH stainless steel head caps that were supposed to have been heat treated according to specification AMS- 5355D, condition H1000. The H1000 refers to the ageing temperature, in this case 1000°F. The minimum hardness required by specification AMS-5355D, 1000H for this ageing temperature is HRC 34. The AMS specification gives homogenization, solution, and ageing temperatures and times at which the 17-4 PH stainless steel is to be heat treated. It also gives the minimum hardness corresponding to the ageing temperatures. The specification does not give representative microstructures corresponding to the recommended heat treatments. To determine if the head caps submitted for evaluation had been given the proper heat treatment, heat treatments including homogenization, solution anneal, and ageing at 925°F, 1025°F, 1075°F, and 1150°F were performed for comparison with the as-received material. Additional samples were solution annealed, aged at 925°F ,but not

homogenized. The hardness and microstructures for all of the heat treated specimens were compared with the hardness and microstructures of the as-received head caps.

The chemical analyses, primarily the carbon and silicon determinations, indicated that head caps NIST 1 and 20 were made from different heats of 17-4 PH stainless steel than head caps 5066 and 5441. A comparison of the hardness for all of the as-received head caps to that for the specimens taken from head caps 5066 and 5441 and homogenized, solution annealed, and aged at 925°F, 1025°F, 1075°F, or 1150°F indicated that none of the as-received head caps were heat treated at the ageing temperature of 1000°F where the specified minimum hardness was HRC 34.

The hardness results for samples taken from head cap 5441 that were homogenized, solution annealed, and aged at 925°F, 1025°F, 1075°F, and 1150°F were compared to the as-received hardness (HRC 27.8 ± 1.1) for head cap 5441. The hardness ranged from HRC 39.5 ± 0.4 for the specimen aged at 925°F to HRC 31.1 ± 0.5 for the specimen aged at 1150°F. Ageing the samples taken from head cap 5441 at temperatures from 1025°F to 1075°F produced hardnesses of HRC 32 to 35.

The hardness results for samples sectioned from head cap 5066 and heat treated similarly indicate that head cap 5066 with the as-received hardness of HRC 31.5 ± 0.7 could have been aged at 1150°F where the homogenized, solution annealed, and aged hardness was HRC 31.0 ± 0.6. The specified hardness of HRC 34 was attained in head cap 5066 when it was aged also in the temperature range of 1025°F to 1075°F.

The as-received hardness for head cap NIST 1 was HRC 29.6 ± 2.6. This hardness was similar to the hardness obtained for samples taken from head caps 5066 and 5441 after they were homogenized, solution annealed, and aged at 1150°F, but still less than the specified hardness of HRC 34.

The as-received hardness for head cap 20 was HRB 98.8 ± 5 which corresponds to a HRC of approximately 20, substantially less than the specified hardness of HRC 34.

The microstructures present in the samples taken from head caps 5066 and 5441 that were homogenized, solution annealed, and aged at 925°F, 1025°F, or 1075°F were not the same as the microstructures observed in the as-received head caps 5066, 5441, or 20.

The microstructures observed in samples that were taken from head caps 5066 and 5441 and homogenized, solution annealed, and aged at 1150°F, appeared to be similar, but not the same as that seen in the as-received head caps 5066, 5441, and 20. This microstructure may be the result of a phase transformation that occurred when the steel was aged at 1150°F. The martensite matrix of a homogenized, solution annealed, and aged 17-4 PH stainless steel may begin to revert to austenite when heated in a temperature range of 1130 to 1190°F (4). Hence when the samples in our test program were aged at 1150°F for four hours the homogenized and solution annealed

microstructure began to transform to the microstructure shown in Figures 15a and 15b.

Examination of as-received head cap NIST 1 (Figure 5) showed that the microstructure in this as-received sample was similar to that of a homogenized and solution annealed material. This was concluded primarily from the presence of the matrix of martensite and ferrite platelets. The microstructure most similar to that observed for head cap NIST 1 would be that of head cap 5441 after it was homogenized and solution annealed, but not aged (see Figure 11b). The microstructures appeared to be similar, and the hardness (HRC 29.6 ± 2.6) for as-received head cap 5441 was comparable to the hardness (HRC 31.8 ± 0.9) obtained for the homogenized and solution annealed sample.

The microstructure present in the samples taken from as-received head caps 5066 and 5441 that were solution treated (no homogenizing treatment) and aged at 925°F indicated that there was a lack of homogeneity in the microstructure. It appeared from the microstructures shown in Figures 18 and 19 that the steel had not been subjected to a high enough temperature and time necessary to permit a complete solution of the elements necessary to produce the desired microstructure (see Figure 14). Even though the microstructure in this sample appeared inhomogeneous compared to the other samples examined, the hardness for head caps 5066 and 5441 (HRC 38.0 ± 1.4 and 39.3 ± 0.5, respectively) were characteristic of aged samples.

CONCLUSION:

Metallographic and hardness results indicate that the head caps identified as 5066, 5441, NIST 1, and 20 were not heat treated according to AMS specification 5355D, in particular to condition H1000.

References:

1) AMS Specification 5355D, Society of Automotive Engineers, 1985.

2) ASM Metals Handbook, Heat Treating, Cleaning and Finishing, Vol. 2, page 250, 1964.

3) Aerospace Structural Metals Handbook, Ferrous Alloys, Code 1501, 17-4 PH, Author: J.R. Kattus, March 1978.

4) Armco, 17-4 PH Stainless Steel Bar and Wire, Armco Steel Corp., 1966).

Table 1. Requirements of Specification AMS-5355D.

Chemical Composition
(wt. %)

C	Mn	Si	P	S	Cr	Ni	Nb + Ta	Al	Cu	Sn	N
.06 max	.70 max	.50 to 1.00	.040 max	.030 max	15.50 to 16.70	3.60 to 4.60	.15 to .40	.05 max	2.80 to 3.50	.02 max	.05 max

Hardness (HRC), min.

Condition	
H900	40
H925	40
H1000	34
H1100	32

Table 2. Heat Treatments Specified by AMS-5355D.

Homogenization Treatment:

2100°F ± 25°F, 90 minutes, and air cooled.

Solution Treatment:

1900°F ± 25°F, 60 minutes, and air cooled.

Ageing Treatment:

Condition	Temperature	Time
H900	900°F ± 10°F	90 min. ± 15
H925	925°F ± 10°F	90 min. ± 15
H1000	1000°F ± 10°F	90 min. ± 15
H1100	1100°F ± 10°F	90 min. ± 15

Table 3. Special Heat Treatments of Head Caps numbers 5066 and 5441.

Group 1.

Homogenization:

2150°F ± 25°F, 2 1/4 hours at temperature, air cooled.

Solution treatment:

1900°F ± 25°F,1 hour at temperature, air cooled.

Ageing treatments:

1) No age

2) 925°F ± 15°F, 4 hours, air cooled

3) 1025°F ± 15°F, 4 hours, air cooled

4) 1075°F ± 15°F, 4 hours, air cooled

5) 1150°F ± 15°F, 4 hours, air cooled

Group 2.

Solution treatment:

1900°F ± 25°F, 1 hour at temperature, air cooled.

Ageing treatments:

1) No age

2) 925°F ± 15°F, 4 hours, air cooled

3) 1025°F ± 15°F, 4 hours, air cooled

Table 4. Chemical Composition

Element:	Specification AMS 5355D (wt.%) min...max	5066E	5066R	5441E	5441R	NIST1E	NIST1R	20R	20E
C	- 0.06	.041	.041	.040	.041	.024	.026	.041	.036
Mn	-- 0.70	.33	.33	.30	.29	.30	.31	.30	.29
Si	0.50-1.00	1.01	1.00	.90	.90	.45	.46	.83	.82
P	-- 0.040	.032	.032	.034	.034	.022	.024	.039	.035
S	-- 0.030	.001	.001	.002	.002	.005	.006	.0014	.0015
Cr	15.50-16.7	15.6	15.6	15.5	15.5	15.8	15.8	15.60	15.50
Ni	3.60-4.60	4.05	4.08	4.04	4.0	4.25	4.30	4.11	4.07
Nb plus Ta	0.15-0.40	.18 ND*	.18 ND	.19 ND	.18 ND	.21 ND	.23 ND	.18 ND	.17 ND
Al	-- 0.05	.033	.032	.031	.030	<.01	<.01	.022	.018
Cu	2.80-3.50	3.30	3.32	3.39	3.38	3.48	3.60	3.35	3.27
Sn	-- 0.02	ND	ND	ND	ND	ND	ND	ND	ND
N	-- 0.05	ND	ND	ND	ND	ND	ND	ND	ND

* Not determined

Table 5. Hardness, Rockwell C, for head caps 5066 and 5441

Group 1.

	Head Cap 5066	Head Cap 5441
	Average Std. Dev.	Average Std. Dev.
As-received:	29.4 ± 0.4	28.0 ± 0.5
2150°F,Homogenization:	35.8 ± 1.0	37.7 ± 1.6
1900°F,Solution anneal:	36.8 ± 0.6	31.8 ± 0.9
925°F, Age:	39.6 ± 0.4	39.5 ± 0.4
1025°F, Age:	35.0 ± 0.2	34.5 ± 0.4
1075°F, Age:	33.7 ± 0.5	31.6 ± 0.4
1150°F, Age:	31.0 ± 0.6	31.1 ± 0.5

Group 2*

	Head Cap 5066	Head Cap 5441
	Average Std. Dev.	Average Std. Dev.
As-received:	29.4 ± 0.4	28.0 ± 0.5
1900°F,Solution anneal:	30.8 ± 1.1	36.4 ± 0.8
925°F, Age:	38.0 ± 1.4	39.3 ± 0.5

* These specimens were not homogenized.

Figure 1. Photograph of an as-received head cap, and a section of head cap 20. Arrow A points to the ear, arrow B the ring, and arrow C, the section used for the hardness measurements.

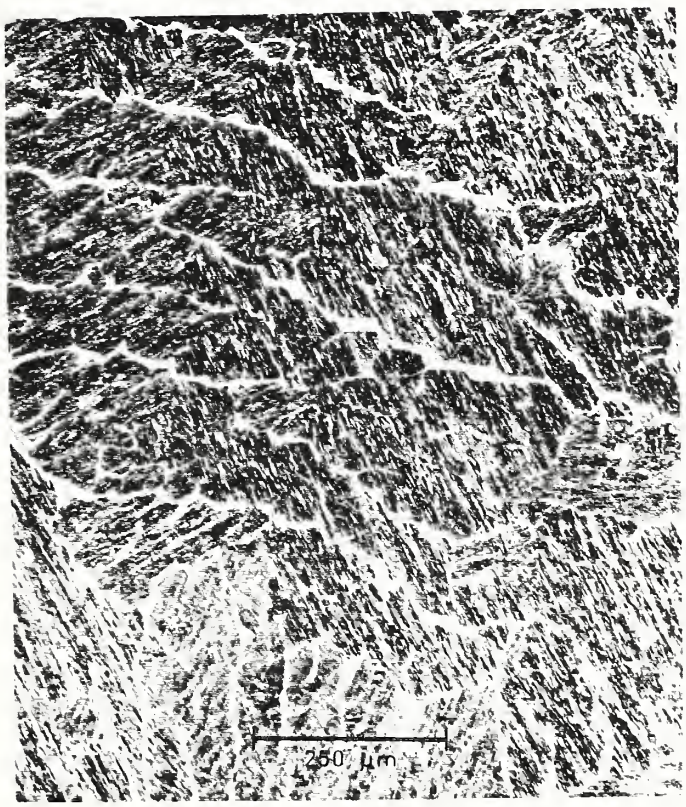


Figure 2. Photomicrographs of as-received head cap 5066.
Magn. X100 Etch: Villela's

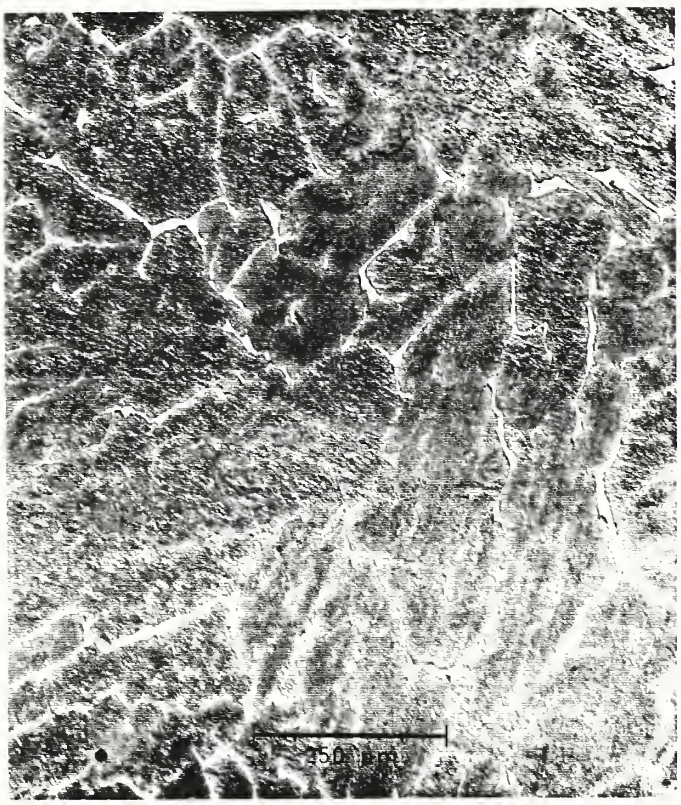


Figure 3. Photomicrograph of as-received head cap 5441.
Magn. X100 Etch: Villela's

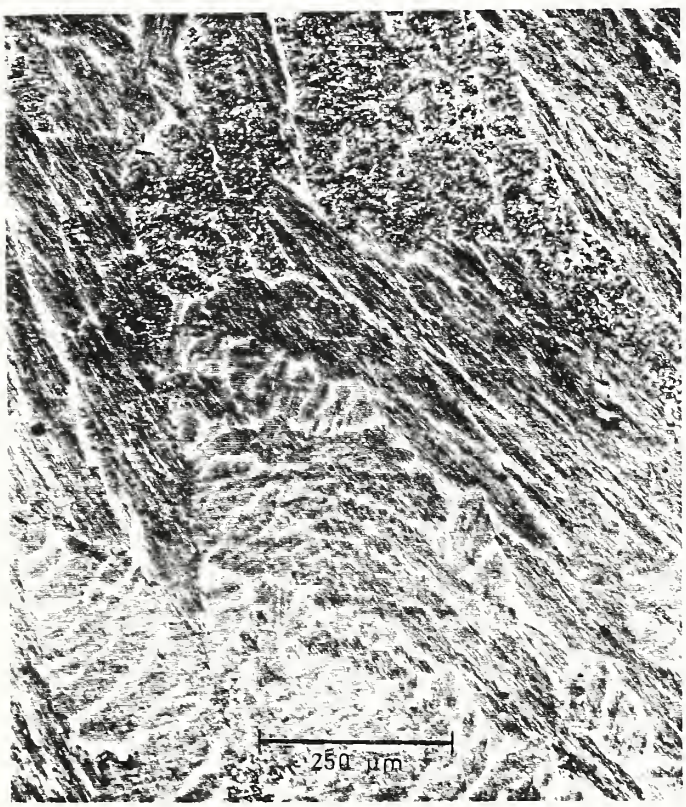


Figure 4. Photomicrograph of as-received head cap 20.
Magn. X100 Etch: Villela's

Figure 5. Photomicrograph of as-received head cap NIST 1.
Magn. X100 Etch: Villela's

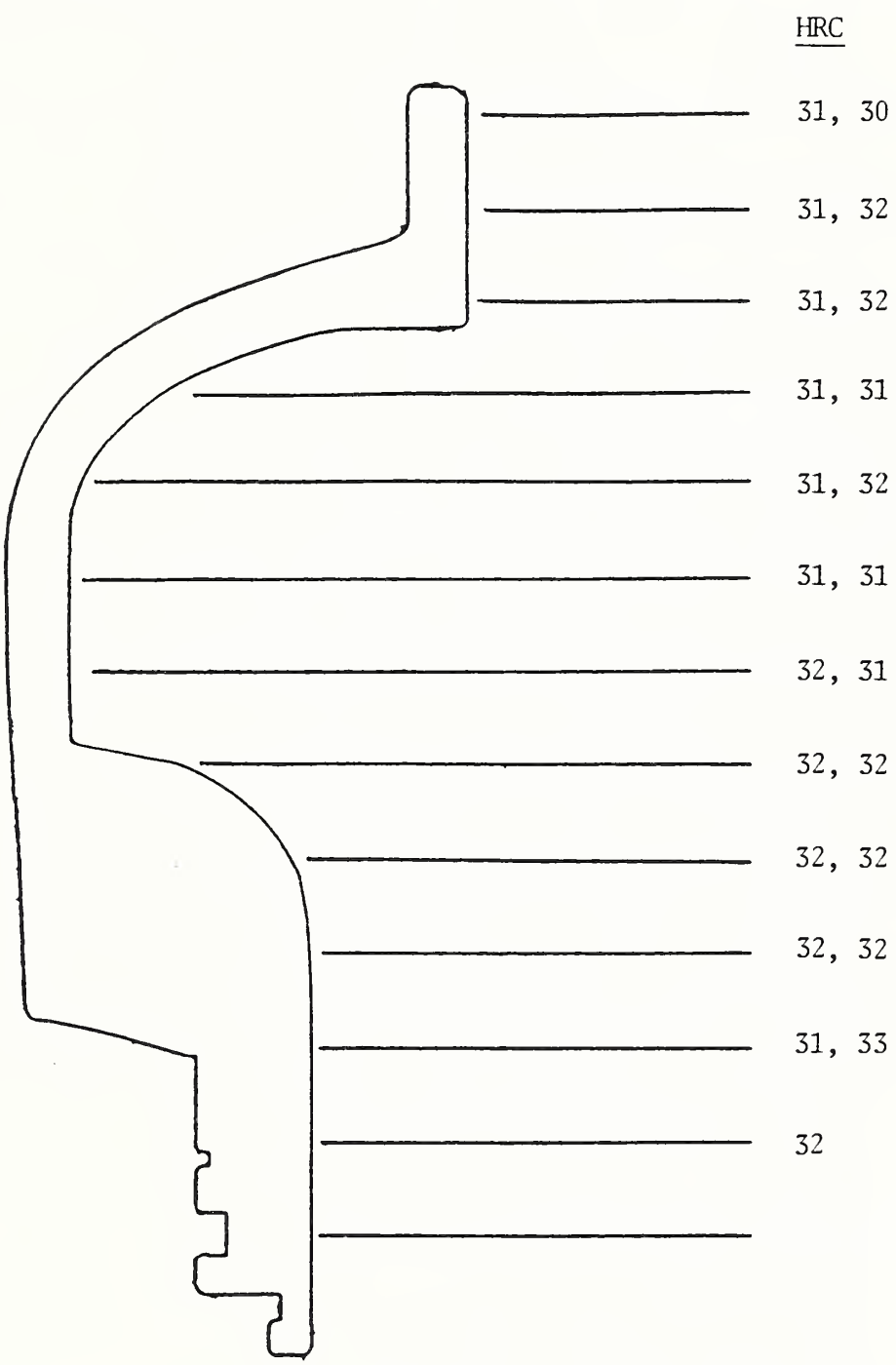


Figure 6. Hardness profile for as-received head cap 5066.

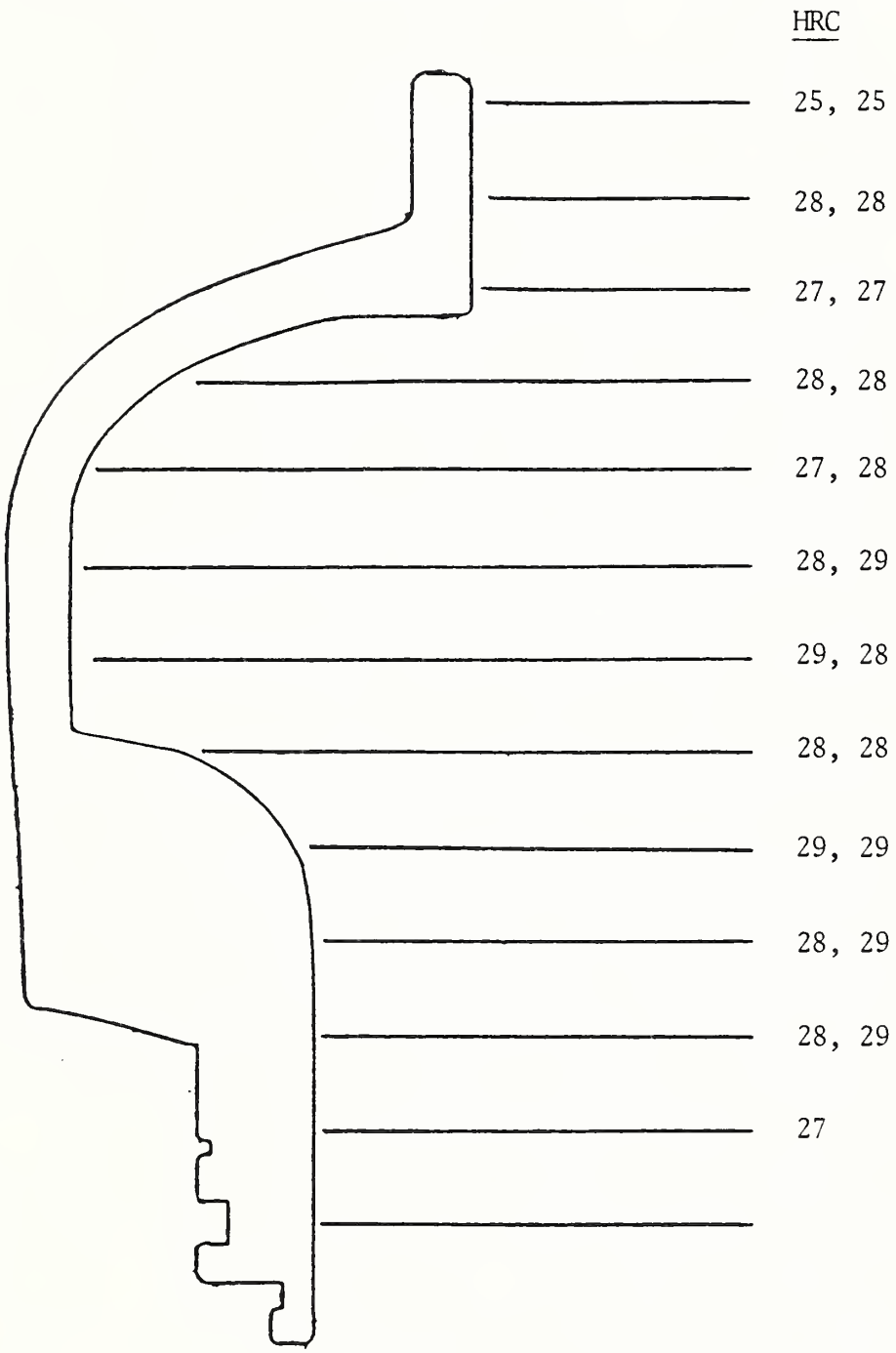


Figure 7. Hardness profile for as received head cap 5441.

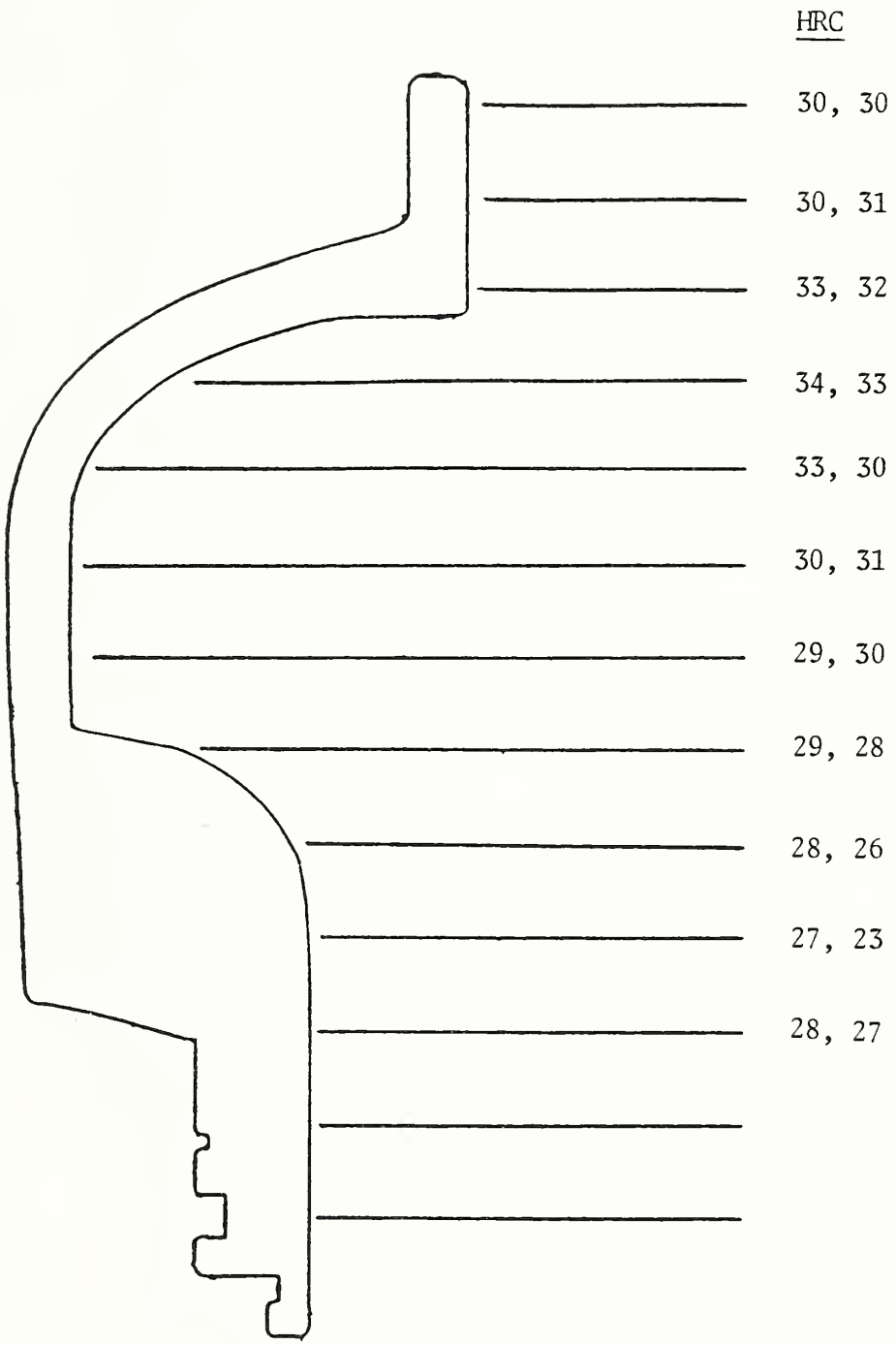


Figure 8. Hardness profile for as-received head cap NIST 1.

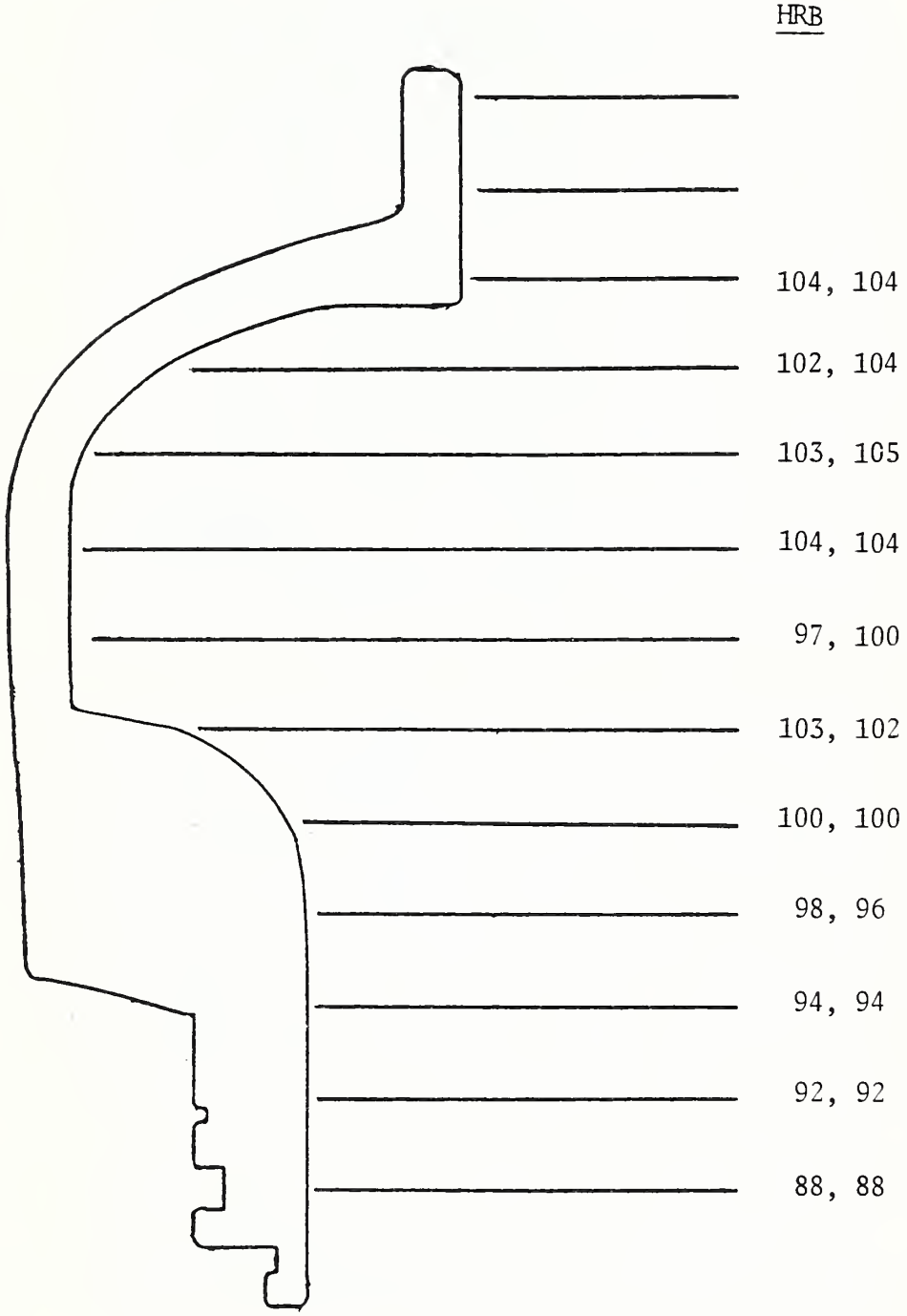
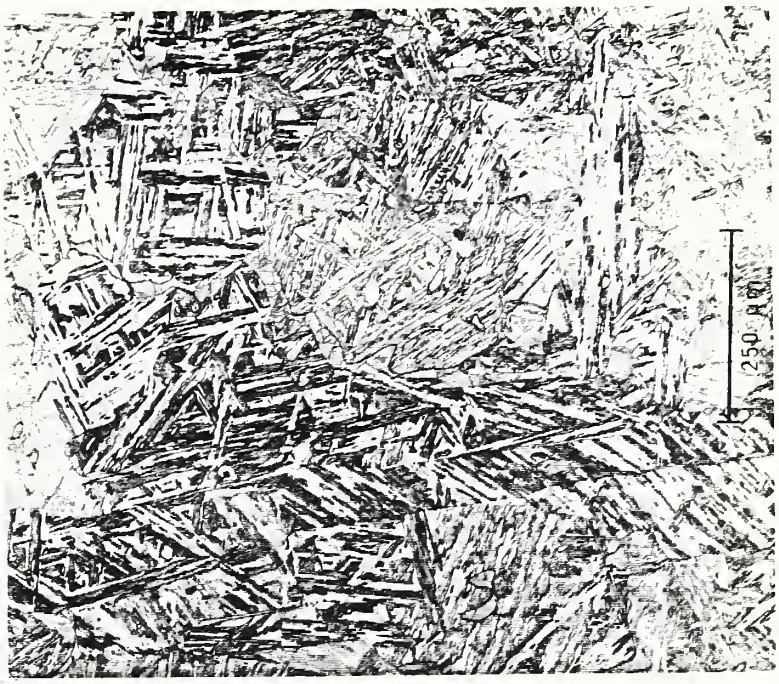
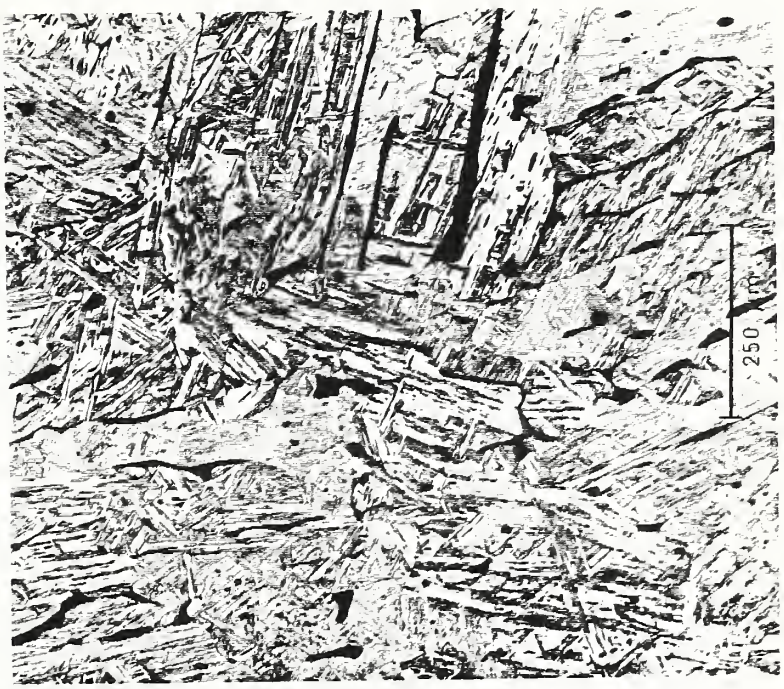


Figure 9. Hardness profile for as-received head cap 20.

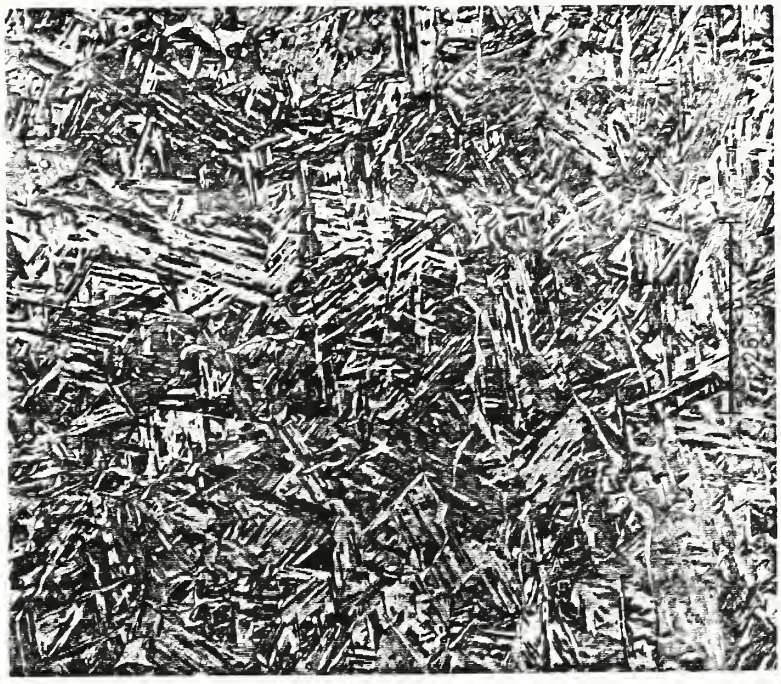


(a)

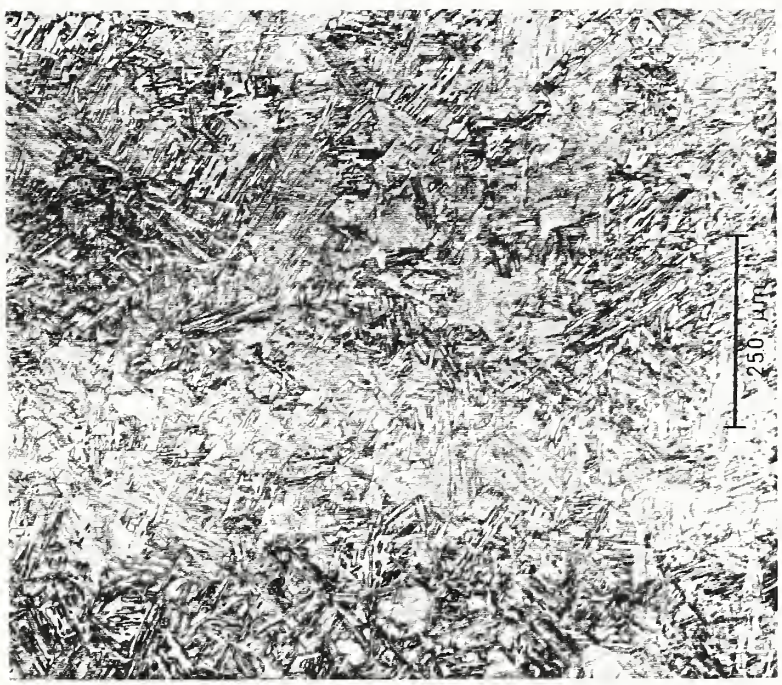


(b)

Figure 10. Photomicrographs of samples taken from head caps 5066 (a) and 5441 (b) after they were homogenized at 2150°F for two and one-quarter hours and air cooled.
Magn. X100 Etch: Villela's



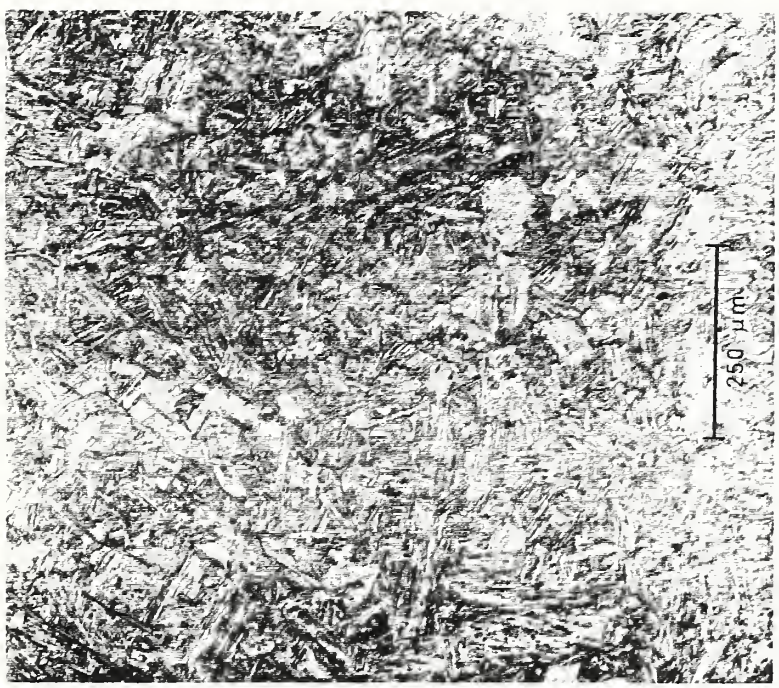
(a)



(b)

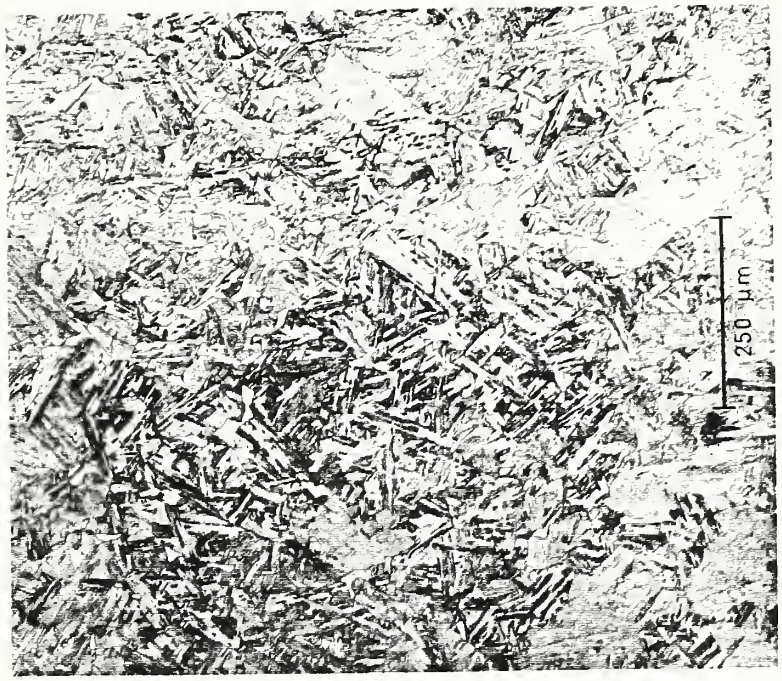
Figure 11. Photomicrographs of samples taken from head caps 5066 (a) and 5441 (b) after they were homogenized, and solution annealed at 1900°F for one hour and air cooled.
Magn. X100 Etch: Villela's

(a)



(b)

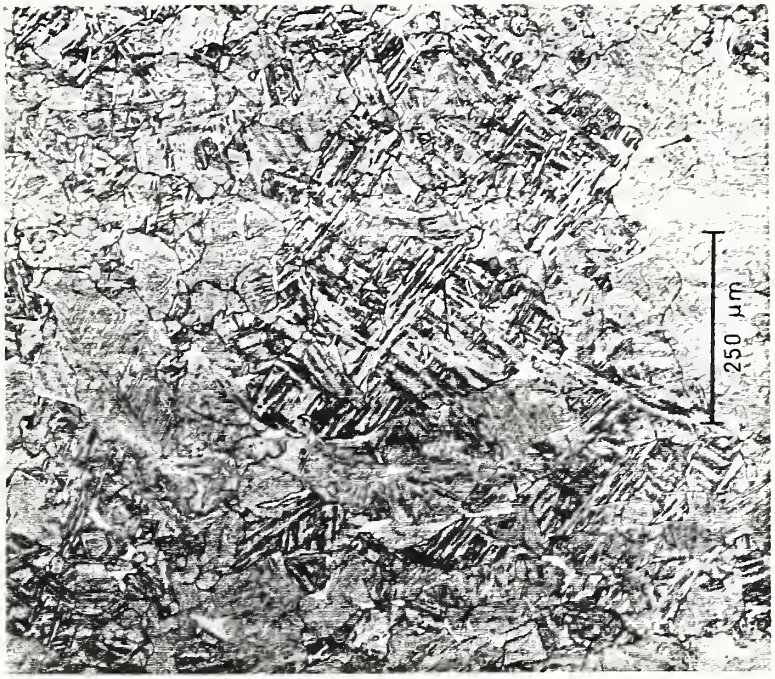
Figure 12. Photomicrographs of samples taken from head caps 5066 (a) and 5441 (b) after they were homogenized, solution annealed, aged at 925°F for four hours and air cooled.
Magn. X100 Etch. Villela's



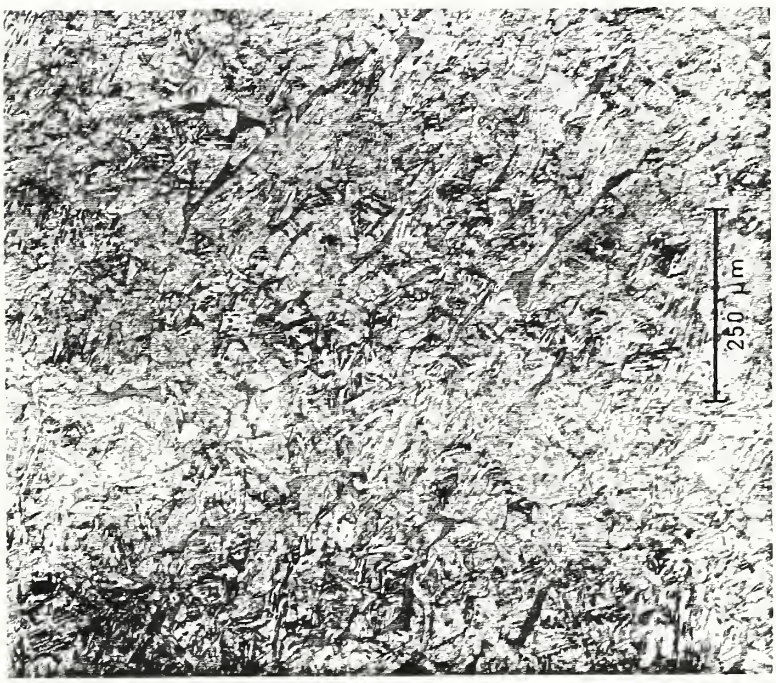
(a)

(b)

Figure 13. Photomicrographs of samples taken from head caps 5066 (a) and 5441 (b) after they were homogenized, solution annealed, aged at 1025°F for four hours and air cooled.
Magn. X100 Etch. Villela's

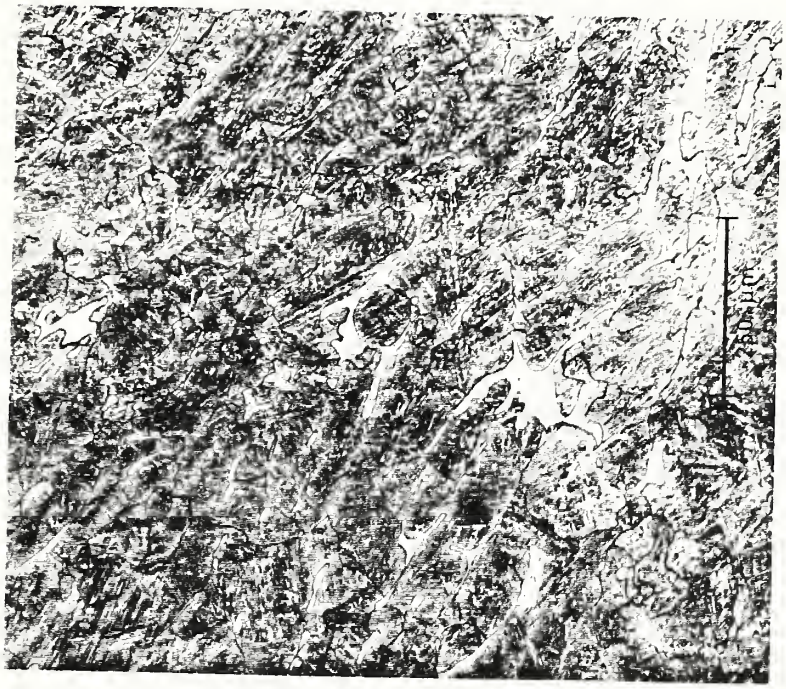


(a)



(b)

Figure 14. Photomicrographs of samples taken from head caps 5066 (a) and 5441 (b) after they were homogenized, solution annealed, aged at 1075°F for four hours and air cooled.
Magn. X100 Etch. Villela's



(a)

(b)

Figure 15. Photomicrographs of samples taken from head caps 5066 (a) and 5441 (b) after they were homogenized, solution annealed, aged at 1150°F for four hours and air cooled.
Magn. X100 Etch. Villela's

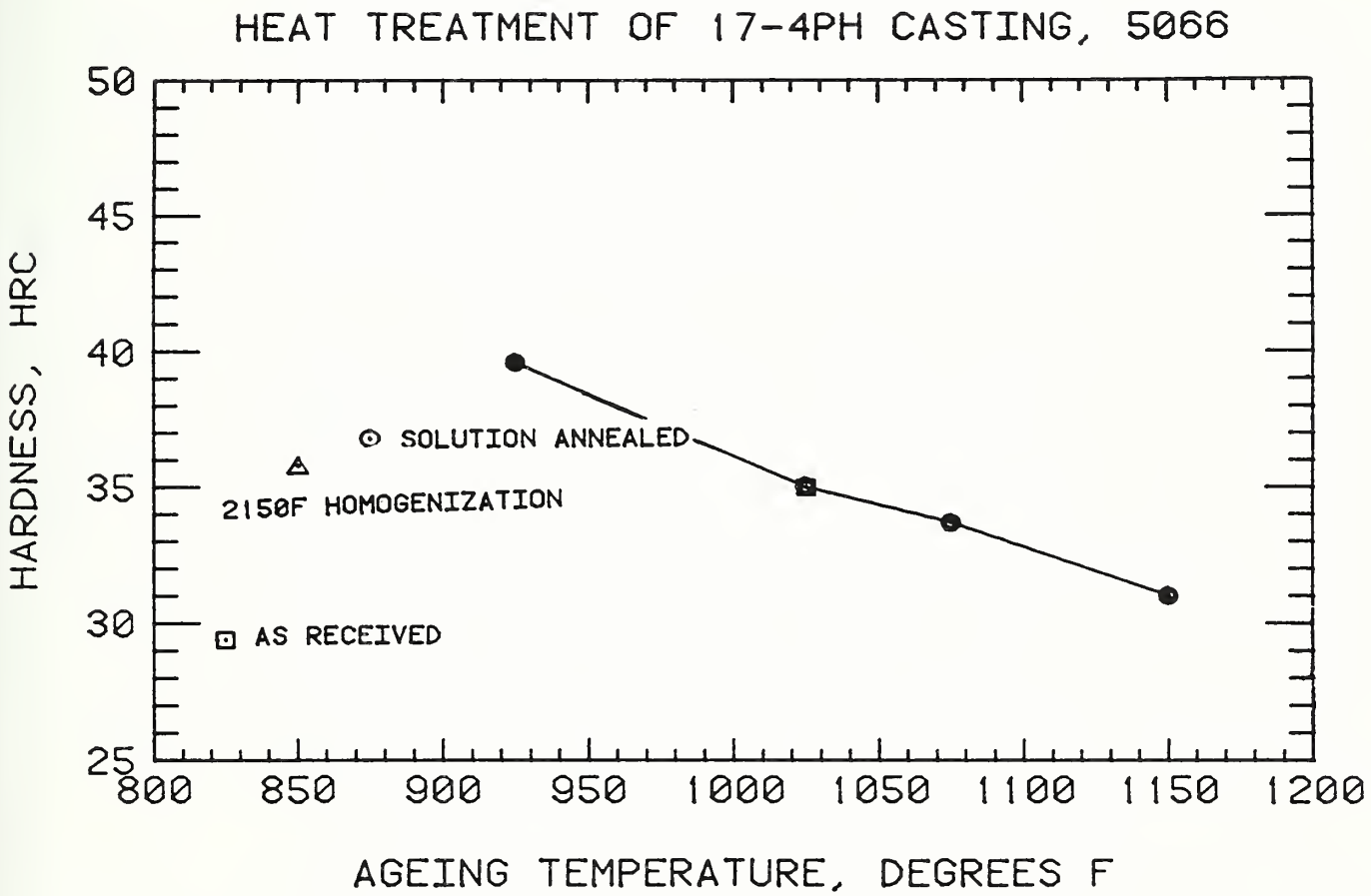


Figure 16. Hardness Rockwell C versus ageing temperature for head cap 5066. Also shown are the hardness values for the as-received, the homogenized, and solution annealed samples.

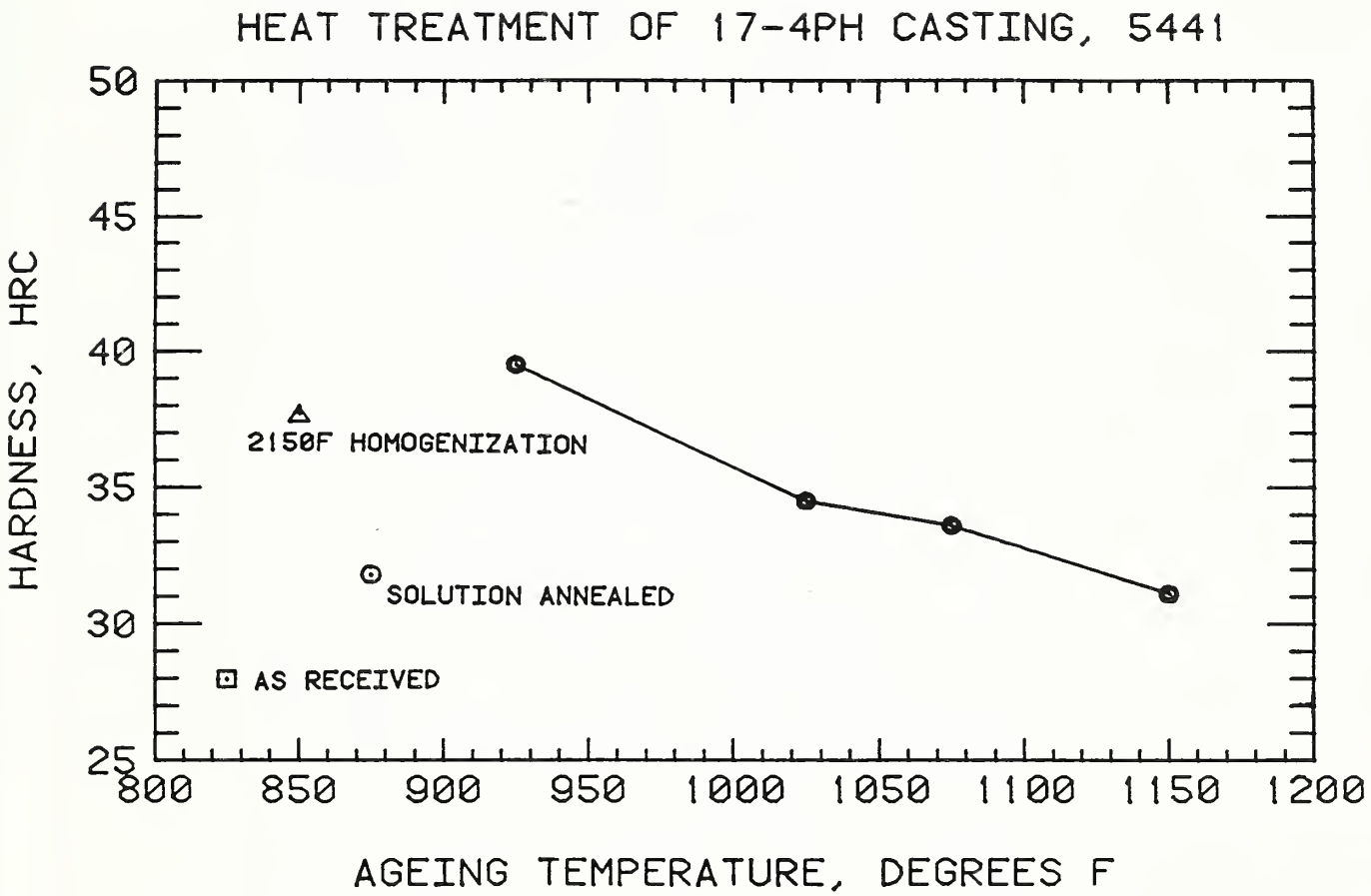
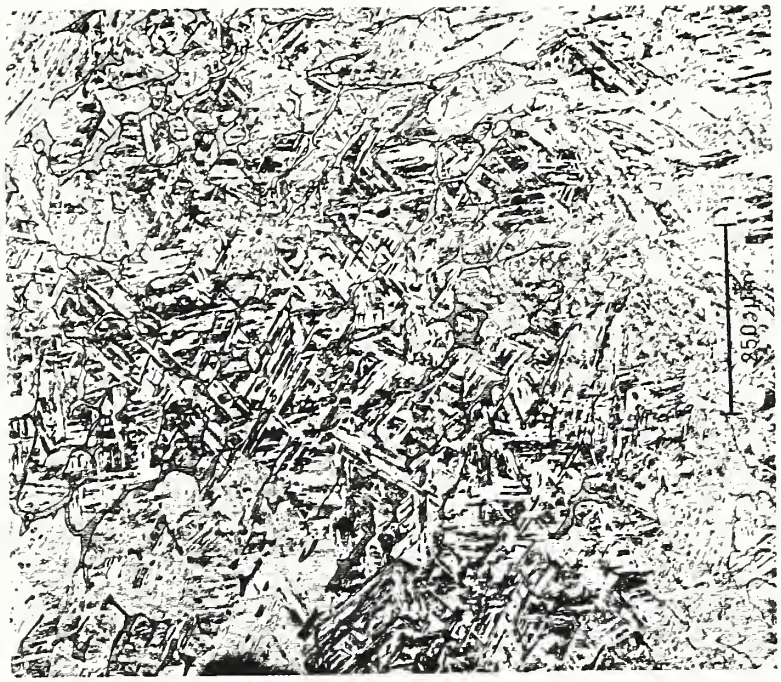
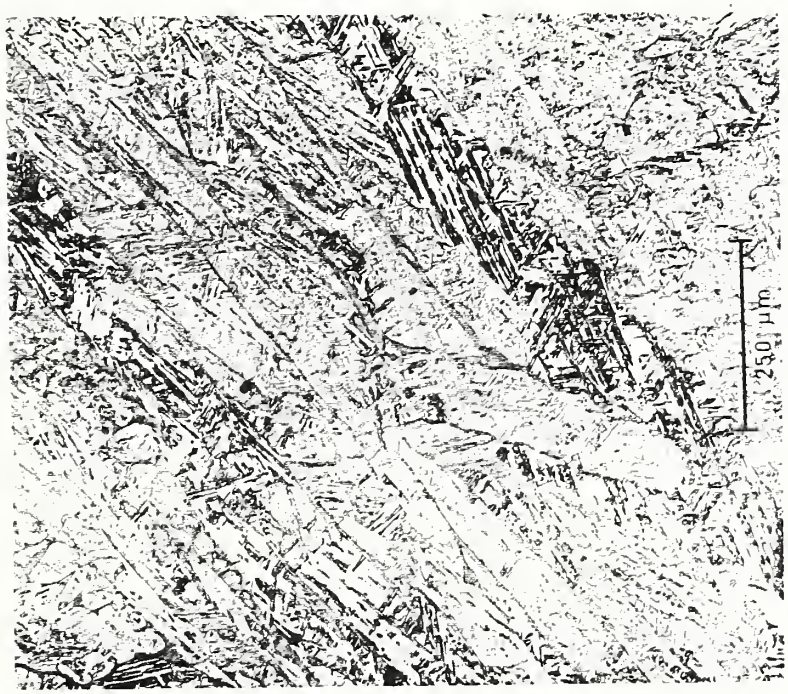


Figure 17. Hardness Rockwell C versus ageing temperature for head cap 5441. Also shown are the hardness values for the as-received, the homogenized, and solution annealed samples.



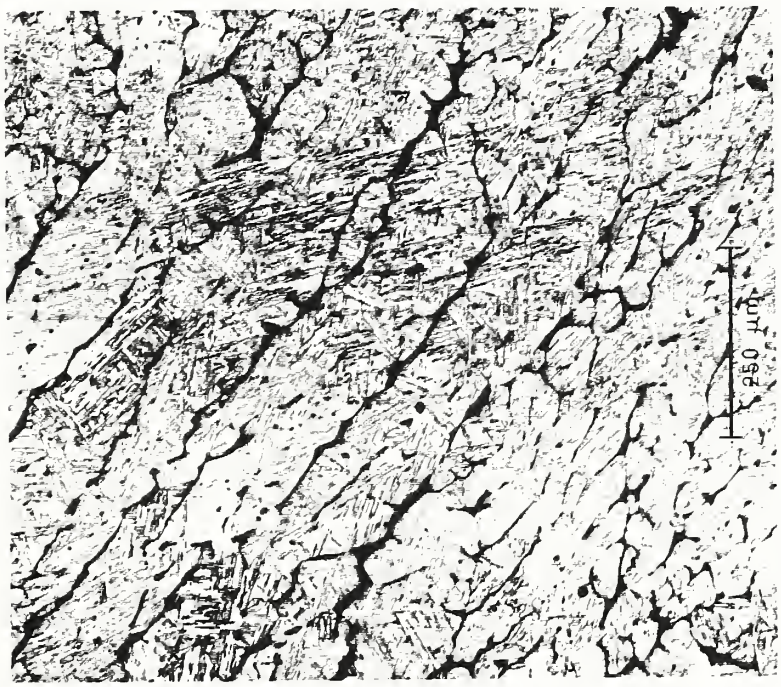
(a)



(b)

Figure 18. Photomicrographs of samples taken from head cap 5066 (a) and 5441 (b) after they were solution annealed at 1900°F for one hour and air cooled. Samples were not homogenized.
Magn. X100 Etch. Villela's

(a)



(b)

Figure 19. Photomicrographs of samples taken from head caps 5066 (a) and 5441 (b) after they were solution annealed, and then aged at 925°F for four hours and air cooled. These samples were not homogenized. Magn. X100 Etch. Villela's

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11. ABSTRACT (A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here)

A metallurgical evaluation was conducted to determine if selected castings of 17-4 PH stainless steel had been properly heat treated as required by SAE specification AMS-5355D. Optical metallographic analysis and hardness measurements were made on four samples of as-received castings and on selected samples that were homogenized, solution annealed and aged at various temperatures.

12. KEY WORDS (Six to twelve entries; alphabetical order; capitalize only proper names; and separate key words by semicolons)

17-4 PH Stainless steel; heat treatment, metallurgical evaluations; stainless steel

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